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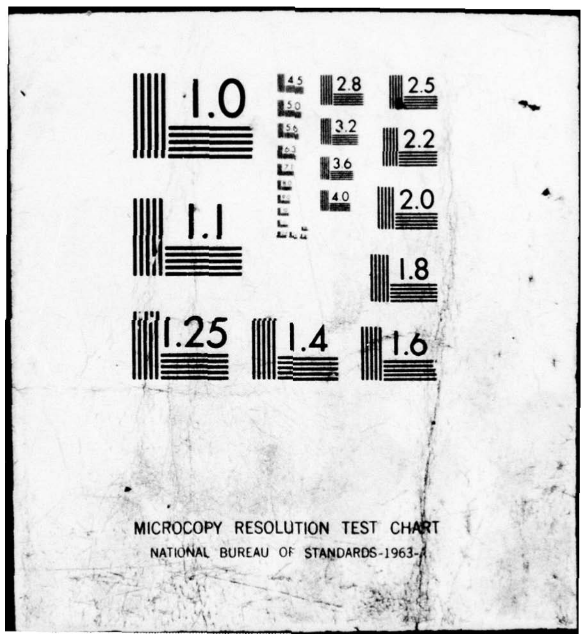
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INTERFACE SEPARATION-LUBRICATION SUBSTANCES FOR ISOTHERMAL FORGING AT 1300F TO 1500F

WESTINGHOUSE ELECTRIC CORPORATION
ADVANCED ENERGY SYSTEMS DIVISION
PITTSBURGH, PENNSYLVANIA 15236

APRIL 1977

TECHNICAL REPORT AFML-TR-77-87
Final Report for Period 9 January 1976 to 15 February 1977

Approved for public release; distribution unlimited.

AIR FORCE MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

NOTICE

This technical report has been reviewed and is approved.

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A program to develop and formulate separation-lubrication compositions for isothermal forging of beta titanium alloys in the temperature range from 1300F to 1500F was successfully completed. Several hundred compositions were chosen at the start of the program, mostly of the vitreous-phase-particulate type, containing boundary film additives or stable semi-abrasives. The field was narrowed to 28 low friction (LFC) types, 12 controlled friction (CFC) types and 8 molten salt (MSC) types. These were further evaluated through quantitative

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measurement of their application, fusion, stability, densification, accumulation and adhesion characteristics.

Optimum combinations of these factors were selected through a simplified multifactor desirability analysis and eight final compositions were selected for further evaluation of their friction reducing properties through ring compression forging at the Westinghouse hot die isothermal forging facility at Wright-Patterson Air Force Base. These compositions included three of the LFC type, one CFC type and one molten salt type for service at the low end of the temperature range (near 1350F) on ferrous alloy dies, and three LFC type for service on nickel alloy dies at the high end of the thermal range (near 1500F).

FOREWORD

This report was prepared by TRW, Inc., Cleveland, Ohio under Westinghouse Electric Corporation Order Number 59-FZK-31010. The overall effort is included under USAF Contract Number F33615-74-C-5059 with Mr. F. J. Gurney as Westinghouse principal investigator. The Air Force Contract was initiated under Project Number 7351, "Metallic Materials", Task Number 735108, "Processing of Metals". The Air Force Contract was administered under the direction of the Metals and Ceramics Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio with Mr. A. M. Adair (AFML/LLM) as the Air Force Project Engineer.

This report covers work performed from January 9, 1976 to February 15, 1977 at TRW, Inc., Cleveland, Ohio. Dr. W. D. Spiegelberg of TRW, Inc. was the Project Engineer and Mr. C. R. Cook was the Program Manager. The work was performed at TRW, Inc. under the direction of Dr. I. J. Toth. Additional technical assistance on the program was provided by Mr. E. Thomas and Mr. A. Banones.

This report was submitted by the author on February 21, 1977.

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I. INTRODUCTION

Considerable interest has arisen recently in hot die processing at die temperatures between 1300 and 1500°F. One of the major opportunities afforded by processing in this temperature regime is a significant cost advantage in producing airframe structural components from the newly developed "beta stabilized" titanium alloys.

These beta titanium alloys have demonstrated substantial improvements in strength, toughness and deep hardenability [1] over current alpha-beta type alloys. One component in the cost advantage therefore is a means for achieving increased structural efficiency (*reduced weight at equivalent load bearing capacity*) in advanced airframes. A second cost advantage component arises from the high degree of ductility possessed by the beta alloys in the solution annealed condition. This is caused by their "body centered cubic" crystal structure (as opposed to the hexagonal close packed structure of the alpha titanium phase) which provides a multiplicity of modes for easy plastic deformation by crystallographic slip. Because of this high ductility, these alloys are expected to have workability and flow stress levels that permit processing at temperatures in the range of 1300-1500°F by hot die isothermal forging. The flow stress characteristics, for example, have been evaluated and found promising.[2]

Hot die processing has demonstrated its cost benefits for aircraft components on several recent programs.[3, 4] The advantages of improved material utilization factors in expensive and strategic materials such as titanium alloys and the savings involved in reduced machining and machining set-up time are apparent. The advantages of processing at an intermediate temperature, lowered toward the range of aluminum processing provides the possibility of simplified hot die forging systems and yet allows expanded utilization of, for example, segmented dies or multiple ram techniques. These would suffer less complication from thermal expansion mismatch, secondary insulation requirements, and special part handling provisions that are present during processing in the 1600-1800°F range. Thus the cost of hot die processing systems can be lowered, but more important, parts with added complexity can be produced.

Isothermal processing, however, has its particular limitations that distinguish it from less precise conventional processing methods. In particular, a critical need exists for a separation-lubrication substance that reduces friction at the interface between die and workpiece during deformation and also provides effective separation without residue after deformation. This substance also must satisfy a long list of additional demands such as chemical compatibility with workpiece and dies, long term stability during elevated temperature preheating, virtually complete absence of die accumulation, ease of application, simplified removal after processing, long shelf life, environmental inertness and moderate cost. These requirements have been recognized, and development of suitable substances has been accomplished for specific hot die forging applications in the temperature range from 1600-1750°F[5] and for less conventional die materials in service over a broad thermal spectrum.[6] The latter investigation revealed the critical nature of separation-lubrication substance composition on die and preheat temperature. A die temperature change as small as one-hundred degrees shows a profound effect on coating effectiveness with no single coating system capable of satisfactory service over a range of more than about 150°F.

Recognition of this limitation and anticipation of the difficult challenge of achieving reduced friction at working temperatures practical for beta titanium alloy processing provided the basis for this investigation.

II. ALTERNATIVES IN SEPARATION-LUBRICATION SYSTEMS

This section deals with the raw materials utilized in development of advanced separation-lubrication substances and with the rationale behind their selection. The combination of the raw materials in formulations for optimized application to preforms or dies in hot die processing is also discussed.

A. Separation-Lubrication Materials

Several options are available among potential separation-lubrication substances for hot die processing in the temperature range of 1300 to 1500°F. These include glass-based types which have proven excellent in reducing friction, "non-glassy" formulations which have demonstrated fine surface finish control, and metallic, ceramic or hybrid coatings bonded to the hot dies. These alternatives are reviewed in the following paragraphs.

1. Low Friction Coatings

When large reductions are taken in isothermal forging operations, as, for example, when deformation starting with a very simple blocked preform is carried to a near net shape, interfacial shear resistance must be low. Since work done against friction makes up a major portion of the total work done in the process, low interfacial shear stress will permit forging loads low enough to minimize the size of equipment required and will insure dependability of tooling components including the hot dies themselves and structural ceramic insulation.

a. Vitreous Systems

The best way to minimize friction in isothermal (or conventional) forging is to apply a coating of a glass frit or mixture of glass forming oxides to the workpiece. Preheating this coating at a temperature above its softening point (where the glass has a viscosity of $10^{7.6}$ poise) causes the material to flow under its own weight. Surface tension forces then take over and cause formation of a uniform but viscous film over the entire coated surface. Figure 1 illustrates the viscosity-temperature behavior of a few selected glass forming compositions based primarily on published[7,8,9] composition-viscosity compilations.

The reasons for specifying a "glass forming" composition rather than a more general "molten salt" system involves the elevated temperature viscosity behavior. Glass forming substances in general are those whose viscosity at the liquidus temperature is 100 poise or greater. Nearly all "molten salts" (including liquid water) have viscosities not far different from 0.01 poise at their liquidus temperature.[10]

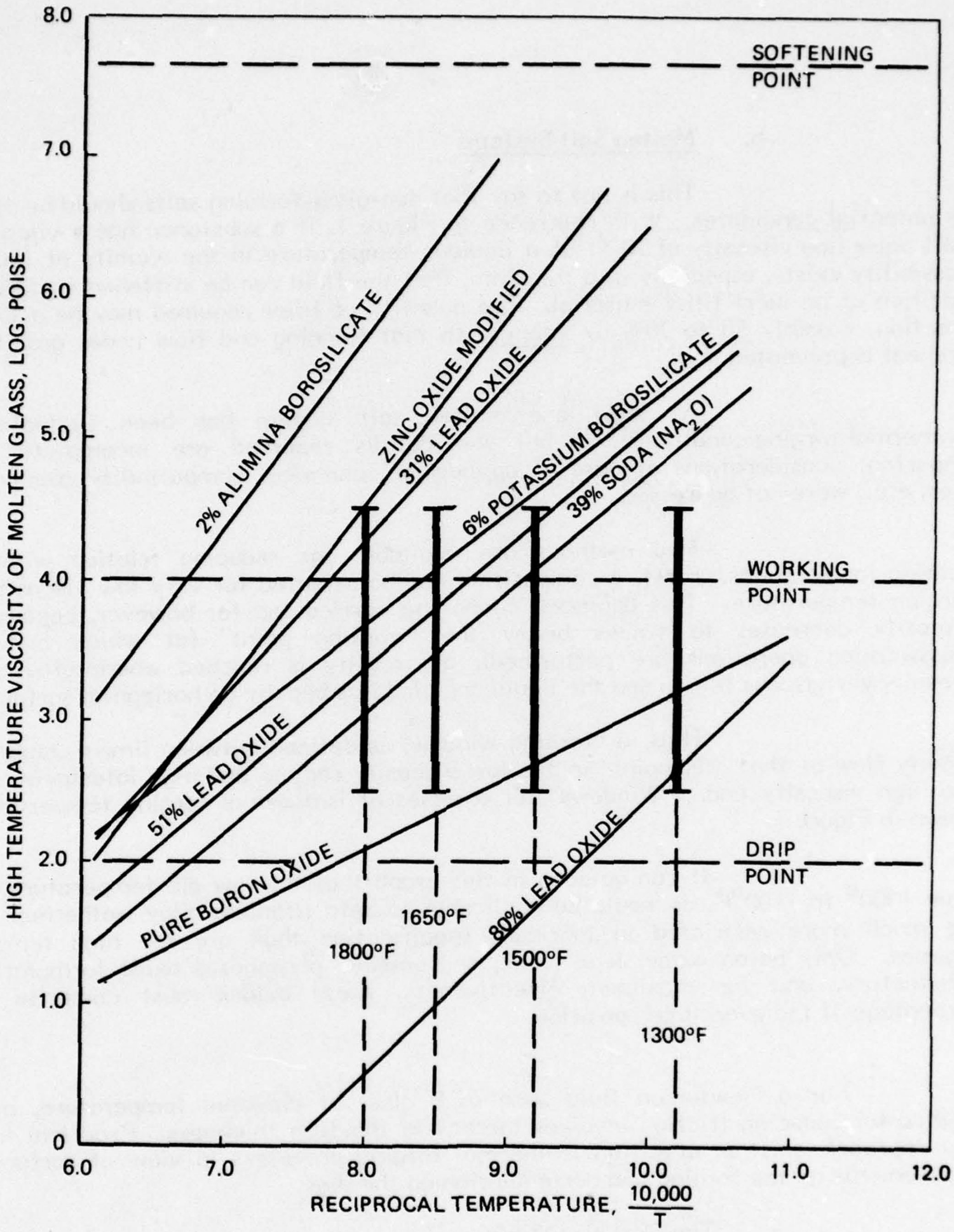


Figure 1. Log Viscosity Versus Reciprocal Temperature For Selected Glass-Forming Compositions.

b. Molten Salt Systems

This is not to say that non-glass-forming salts should be eliminated as potential candidates. With reference to Figure 1, if a substance has a viscosity near 0.01 poise (log viscosity of 10^{-2}) at a liquidus temperature in the vicinity of 1300°F, the possibility exists, especially in a thin film, that the fluid can be stiffened significantly by addition of an inert filler material. The quantity of filler required may be a substantial fraction, possibly 50 to 70% by volume, so that thinning and flow under gravity during preheat is prevented.

At least one molten salt system has been evaluated under isothermal forging conditions[11], but the details reported are incomplete and the important considerations of ease of application, chemical compatibility, removal from dies, etc., were not addressed.

Two methods are available for reducing friction with viscous coating formulations. First, a composition can be selected for very low viscosity at the forging temperature. This approach cannot be carried too far however, because as the viscosity decreases to values below the "working point" (at which conventional glassworking operations are performed), a viscosity is reached where gravity forces prevail over viscous forces and the liquid accumulates heavily on horizontal surfaces.

Thus, a "forging window" is defined between limits established by gravity flow at the "drip point" in the low viscosity regime and high interfacial shear at the high viscosity end. "Windows" for a series of isothermal forging temperatures are shown in Figure 1.

It can be seen in this graph that the low die temperature regimes from 1300° to 1500°F, as would be applicable to beta titanium alloy isothermal forging, are much more restricted in lubricant specification than are the high temperature regimes. Only boron oxide, lead oxide, and possibly phosphorus oxide formulations are satisfactory, and for maximum effectiveness, these oxides must comprise a high percentage of the overall composition.

For a Newtonian fluid such as a glass at elevated temperature, a second method for reducing friction involves increasing the film thickness. Practical limits to this approach exist in precision isothermal forging processes in view of surface finish requirements on the forging and accumulation on the dies.

c. The Partition Ratio

During a forging operation, a certain "partition ratio" is maintained. That is, on removal of the part from the die, a certain fraction of the original coating remains behind. For the next piece, the effective interface coating thickness includes both the residual and new coatings, and again the partition ratio determines the distribution between workpiece and die. After several parts are forged, a uniform film builds on the die with a thickness determined by the coating thickness and partition ratio, as well as the glass viscosity, and local temperature conditions.

The behavior with time of this partitioning is illustrated in Figure 2. This graph illustrates the phenomenological concepts. For example, there is a certain "conditioning" or "break-in" period for the isothermal forging dies during which the film thickness builds to a steady state value. Both the die conditioning period and the steady film thickness are determined by:

a) coating effects, such as viscosity, degree of wetting on the die and degree of bonding to the workpiece;

b) thermal factors such as die and preheat temperatures; and,

c) die and workpiece surface finish and condition.

In isothermal forging where both workpiece and die are at the same temperature, the partition ratio must be near 50%. This can be modified in practice with die coatings that reduce wetting by the glass, by wetting agents in the glass composition that act more efficiently on titanium than on the nickel alloy dies, or more effectively, by a combination of titanium alloy wetting agents and an inert separating compound mechanically mixed into the glass powder.

The lubrication-separation substance development in earlier work[6] was aimed at reducing the partition ratio through addition of particulates to assist the separating action. The glass compositions in general were alkali free borosilicates with the transition metal oxides as wetting agents. These formulations were developed for use at relatively high die temperatures with a variety of die materials including a ceramic, namely, silicon nitride. Emphasis on the present work has been directed toward the 1300-1500^{o}F range.

Table I contains a list of materials utilized on this program with data concerning chemical and physical characterization. The vitreous phase "matrix" compositions consisted of two with specified composition and two commercial grades as listed in Sections I and V of this table. Controlled low viscosity was the prime reason for the composition selection in all cases since it was felt that reactivity was not as serious a concern at the relatively low temperature of the intended processing.

The second section of the table deals with particulates in two general classes, namely lamellar solids and semi-abrasive particles.

Molten salt compositions were included in the investigation since, although their viscosity is expected to be low at the liquidus temperature, some advantage might be found with operation in the "mushy" freezing zone between the liquidus and solidus in a composition modified to be off-eutectic.

Some other molten salts, namely sodium silicate and sodium phosphate, are listed under Section V in Table I. These were used as inorganic binders and as low friction coatings.

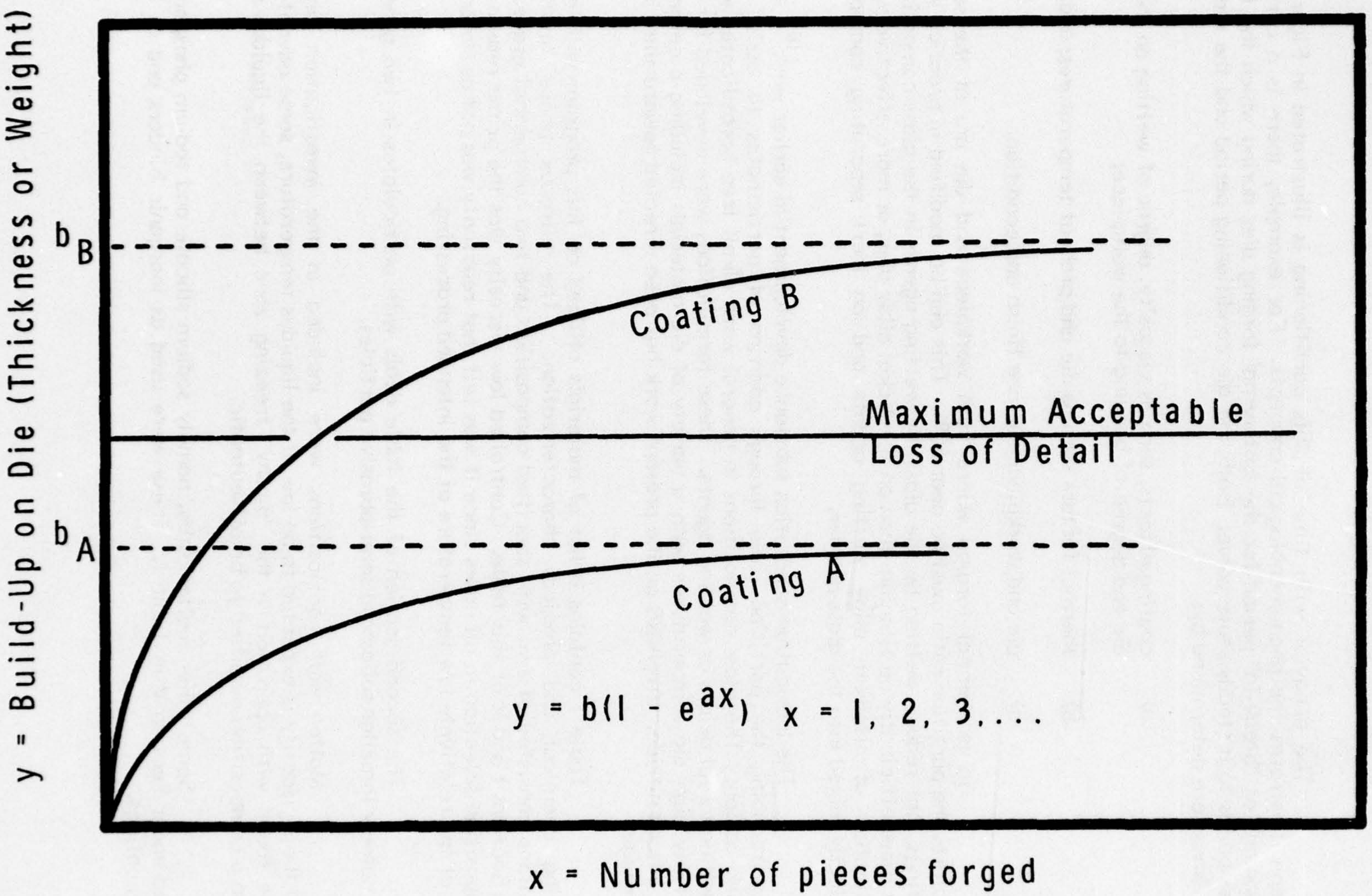


Figure 2. Buildup of Film Thickness on Hot Die Showing Transient and Steady State Conditions.

TABLE I

COMPOSITION OF COATING, WORKPIECE AND DIE MATERIALS

I. VITREOUS PHASE (Weight Percent)

38BB	22A
60 B_2O_3	42 SiO_2
31 SiO_2	2 Na_2O
7 K_2O	6 K_2O
2 CoO	49 PbO
	1 Li_2O

II. PARTICULATE PHASE

A. Lamellar Solids

Material	Code	Composition	Average Particle Size
Boron Nitride	#1260	Boron 43.61% Nitrogen 56.2% B_2O_3 0.09% Fe 0.01%	1-2 microns
Molybdenum Disulfide	#1492	MoS_2	4 micron
Titanium Diselenide	#1522	$TiSe_2$	5 micron
Calcium Fluoride	#4168	CaF_2 <.01% Cl <.01% SO_4	-325 mesh
Graphite	#1651	C	-200 mesh

TABLE I (contd)

COMPOSITION OF COATING,
WORKPIECE AND DIE MATERIALS

B. Semi-Abrasive Particles

Material	Code	Composition	Average Particle Size
1. Titanium Nitride	89359	TiN (20.6% N_2)	-200 mesh
2. Titanium Carbide	T1276	TiC	3-6 micron
3. Tantalum Carbide	214-6-1	TaC	-200 mesh
4. Tungsten Carbide	WC288	WC	1-5 micron
5. Chromium Carbide	1349	Cr_3C_2	6-8 micron
6. Silicon Carbide	1200	SiC	6-8 micron
7. Aluminum Oxide	173/22025	Al_2O_3	1 micron

III. MOLTEN SALTS

A. Holden experimental Ti-lube #4-6-83 eutectic mixture containing CaF_2-MgF_2-$BaCl_2$.

B. Holden U-5 commercial neutral salt - eutectic mixture of carbonate salts.

Both samples supplied by A. F. Holden Co.

TABLE I (contd)

COMPOSITION OF COATING,
WORKPIECE AND DIE MATERIALS

IV. LIQUID PHASE BINDERS

A. ACR-1

Acrylic Emulsion
50% by weight in xylene

B. DC997

Silicone Varnish
60% by weight in xylene
Lot BHQ 54S

C. Polyimide 2080 Lot No. 10-3-079-9
Supplied by Upjohn Corp.

D. Sodium Silicate Na_2SiO_3

40-42° Bé
50-S-338

E. Sodium Phosphate Na_2HPO_4

Anhydrous Powder S-372

V. COMMERCIAL LUBRICANTS

A. DM254F

Low Temperature Extrusion Lubricant
CHI VIT. CORP.

B. LF22

Lubri-Film Corp.
Coarse graphite in toluene with high temperature binder

C. CRN

Markal Corporation - heat treating coating in aromatic carrier

TABLE I (contd)

COMPOSITION OF COATING,
WORKPIECE AND DIE MATERIALS

VI. TITANIUM ALLOY 38644 - BETA C

	C	N	Fe	Al	V	Cr	Zr	Mo	O	H
0.050 in. Thick Sheet	.01	.012	.06	3.4	8.1	5.6	3.6	4.3	.102	72 ppm
2.50 in. Diam. Bar										
0.125 in. Thick Plate	.02	.011	.06	3.4	8.3	5.8	3.9	4.2	.107	59 ppm

Sheet	RMI Heat 690507	Sol. Anneal 1500°F, 1 Hr, A.C. As Forged
Bar	RMI Heat 600393	Sol. Anneal 1700°F, 30 min. A.C. +1500°F 30 min., A.C.
Plate	RMI Heat 304324	Sol. Anneal 1500°F, 20 min. A.C. +1000°F, 8 Hrs. + 1050°F, 4 Hrs., A.C.

VII. DIE MATERIALS

A. Nickel Base Alloy IN-100 Cast Bar and Plate - TRW Metals Division

Nominal Composition - Weight Percent

C	Cr	Ni	Co	Mo	Ti	Al	B	Zr	V
0.18	10.0	Bal	15.0	3.0	4.7	5.5	0.014	0.06	1.0

B. Iron Base Alloy - A286

Wrought Bar - 2-3/4" Diameter

TRW Heat Code - VEE850

Bar Extruded to 1/2 Inch Diameter for Compatibility Samples.

Nominal Composition

C	Mn	Si	Cr	Ni	Mo	Fe	Ti	Al	B
0.05	1.35	0.50	15.0	26.0	1.3	Bal	2.0	0.2	0.015

2. Controlled Friction Coatings

The term isothermal "sizing" as opposed to isothermal "forging" refers to a reduction taken to bring a forging to final net dimensions and surface finish. Ease of release from the die is vital and accumulation on dies is intolerable for this type of operation. For greatest effectiveness the process should be carried out at the lower end of the temperature range and the higher end of the forging load range within design allowables.

A coating for isothermal sizing (or restriking) does not contain glass except in minor amounts to act as a binder. Several commercial products are potential candidates, for example: Fel Pro C-300, a molybdenum disulfide-graphite formulation; DGF-123, colloidal graphite; LSG 300 containing lead, tin, and volatile organics; and several containing graphite, molybdenum disulfide and boron nitride. Little work has been done with these except for a brief evaluation by ring compression testing at AFML.[12]

The materials utilized in formulation of the controlled friction coating were selected from the lamellar solid and semi-abrasive particles in Table I.

B. Formulations

Table II lists the formulations utilized during the test program. All are presented in terms of a hypothetical 75 gram lot, which represents a convenient volume for coating several coupon type samples. The formulations were designed on the basis of preliminary evaluation of both green and fused coatings at a constant green film thickness of 0.006 inches per side when applied by immersion. Green coatings must possess acceptable durability in handling, and fused coatings must be free from visible defects, non-wetting characteristics, and excessive particulate agglomeration.

The slurry viscosity when mixed with the binder and diluant amounts in the table make an acceptable coating. Scaleup to larger quantities can be performed directly, but some further quality evaluation is advised when making these, both because of batch-to-batch variations in the ingredients and because of potential non-linear mixing effects on slurry control parameters as is occasionally observed in these types of coatings. Formulations may also require some modification (usually dilution) for spray application.

TABLE II

FORMULATION OF LUBRICATION-SEPARATION COMPOUNDS

Coating	Vitreous Phase Type	Vitreous Phase Amount (Grams)	Particulate Type	Particulate Amount (Grams)	Binder Type	Binder Amount (Grams)	Diluant Type	Diluant Amount (Grams)
LFC 1	38BB	50	SiC	2.5	ACR-1	6.7	ISO	15.0
3	"	"	$TiSe_2$	"	"	"	"	"
5	CRN	"	TiO_2	4.0	"	6.4	XYL	10.0
7	38BB	"	TiN	2.5	"	6.0	ISO	7.5
9	"	"	TaC	"	"	"	"	"
11	"	"	Cr_3C_2	"	"	"	"	"
13	"	"	WC	"	"	"	"	"
15	"	"	Al_2O_3	"	"	"	"	"
17	"	"	CaF_2	"	"	5.8	"	5.0
19	"	"	-	"	"	6.0	ISO	10.0
LFC 2	22A	50	SiC	2.5	"	6.2	ISO	10.0
4	"	"	$TiSe_2$	"	"	"	"	"
6	"	"	TiN	"	"	5.8	"	"
8	"	"	TaC	"	"	"	"	"
10	"	"	Cr_3C_2	"	"	"	"	"
12	"	"	WC	"	"	"	"	"
14	"	"	Al_2O_3	"	"	"	"	"
16	"	"	CaF_2	"	"	6.0	"	7.5
18	"	"	-	-	"	6.0	"	10.0
20	"	"	CeO	4.0	"	5.0	"	5.0
22	"	"	TiC	4.0	"	5.0	"	5.0
26	DM254F	50	TiN	3.0	ACR-1	7.8	ISO	15.0
28	"	"	TaC	"	"	"	"	"
30	"	"	Cr_3C_2	"	"	"	"	"
32	"	"	WC	"	"	"	"	"
34	"	"	Al_2O_3	"	"	"	"	"
36	"	"	CaF	"	"	"	"	"
38	"	50	-	-	ACR-1	7.8	"	15.0

All weights in grams - material designations refer to Table I

TABLE II, (contd)

FORMULATION OF LUBRICATION-SEPARATION COMPOUNDS

Coating	Vitreous Phase Type	Vitreous Phase Amount (Grams)	Particulate Type	Particulate Amount (Grams)	Binder Type	Binder Amount (Grams)	Diluant Type	Diluant Amount (Grams)
CFC 1	-	-						
2	-	-	Graphite	40.0	DC997	20.0	XYL	20.0
3	-	-	MoS_2	60.0	DC997	20.0	"	14.0
4	-	-	MoS_2	10.0	PI-2080	50.0	-	-
5	-	-	$TiSe_2$	30.0	ACR-1	10.0	ISO	9.0
6	-	-	$TiSe_2$	30.0	DC997	10.0	XYL	8.0
7	-	-	MoS_2	16.0	ACR-1	16.0	XYL	18.0
	-	-	PbO	16.0				
	-	-	Graphite	20.0				
8	-	-	Graphite	20.0	DC997	7.0	XYL	60.0
9	-	-	"	20.0	ACR-1	7.0	"	60.0
10	MSC-8	5.0	Graphite	5.0			Water	50.0
11	-	-	-	-	PI-2080	50.0	-	-
12	-	-	Graphite	2.5	PI-2080	50.0	-	-

TABLE II (contd)

FORMULATION OF LUBRICATION-SEPARATION COMPOUNDS

	Molten Salt		Particulate		Binder		Diluant	
Coating	Type	Amount (Grams)	Type	Amount (Grams)	Type	Amount (Grams)	Type	Amount (Grams)
MSC-1	U5	40.0	-	-	ACR-1	6.9	ISO	29.0
MSC 2	U5	40.0	Li_2CO_3	2.0	"	7.1	"	29.0
3	U5	40.0	K_2CO_3	2.0	"	7.1	"	29.0
4	U5	40.0	Li_2CO_3	4.0	"	7.3	"	29.0
5	U5	40.0	K_2CO_3	4.0	"	7.3	"	29.0
6	4-6-83	*	-	-	-	-	-	-
7	Sodium Phosphate	*	-	-	-	-	-	-
8	Sodium Silicate	*	-	-	-	-	-	-

*Used in as-received form

III. EVALUATION OF SEPARATION-LUBRICATION SUBSTANCES

An ideal separation-lubrication substance for isothermal processing is one that reduces friction to zero, remains entirely with the finished part after forging with no residual accumulation on the dies, provides assistance in removal of the part from the die, is perfectly compatible with the die and workpiece, and so forth. The list of desirable attributes is long and has been enumerated previously. It is important to recognize that a substance can be found to fulfill any one of these requirements if some of the others are ignored. For example, a thick layer of glass with a viscosity of about 200 poise at the working temperature will reduce friction to a minimum. This approach has not proven practical, however, because accumulation and adhesion are invariably quite high and finished part surface finish and fill are generally unacceptable.

As a second example, a film of a parting compound such as boron nitride can be utilized. This system provides excellent separation, and accumulation difficulties are minimized, but the friction reduction accomplished is small and the achievement of the large plastic deformations required for cost effective isothermal forging of titanium alloys is not possible.

A wide spectrum of material systems exist that should be explored in order to arrive at separation-lubrication substances that best satisfy all requirements simultaneously. Adaptation of vitreous systems through addition of nucleating agents, boundary film modifiers, or soft abrasives is one promising technique; a non-vitreous parting agent approach that concentrates on friction reducing additives is practical; pressure sensitive compounds; and, advanced die applied coatings are only a few of the possibilities. Each system, however, must be rated according to its ability to simultaneously satisfy the many important requirements of separation-lubrication systems.

Arriving at an ideal separation-lubrication system through tailoring the several "response" variables by varying the chemical constituency of coating systems was therefore a major accomplishment of this program. The multi-factor optimization approach utilized and results of its use are described in the following paragraphs.

A. Desirability Assessment Methods

Table III categorizes the quantitative and qualitative evaluation data in a form that allows optimization of separation-lubrication substance "desirability". A numerical rating is assigned to cover the range of each evaluation (response) variable. The factors utilized are:

a) application (visual);

b) weight stability (TGA);

c) thickness stability (densification);

d) die material compatibility (extended exposure);

e) shear adhesion;

f) film accumulation;

g) bulk accumulation; and,

h) localized pitting (short term exposure).

TABLE III - COATING DESIRABILITY FACTOR

	Numerical Rating				
	1	2	3	4	5
APPLICATION	Smooth Uniform Coating Fuses to Glossy Finish	Smooth Uniform Coating Fuses to Matte Finish	Uniform Coating Fuses to Granular Finish	Difficult to Apply Continuous Coating	Cannot Apply Continuous Uniform Coating
STABILITY:					
WEIGHT	+0.23 -0.49	+0.30 +0.59 -0.50 -0.99	+0.60 +0.99 -1.00 -1.49	+1.00 +1.49 -1.50 -1.99	> 1.50 <-2.00
THICKNESS	60 80	81 99 50 59	100 119 40 49	120 149 30 39	> 150 < 30
COMPATIBILITY	0 0.99	1.00 1.99	2.00 2.99	3.00 3.99	> 4.00
ADHESION	0 9.99	10.00 14.99	15.00 19.99	20.00 29.99	>30.00
ACCUMULATION:					
FILM	0 0.99	1.00 1.99	2.00 2.99	3.00 3.99	> 4.00
BULK	No "Extruded" Coating		Slight "Extruded" Coating		Heavy "Extruded" Coating
PITTING	None			Slight Attack on Titanium Alloy	Noticeable Attack on Die Material or Titanium Alloy

The multifactor selection method is based on the concepts developed by Harrington[13] in his "desirability curve", shown schematically in Figure 3. In its conventional form, the desirability curve rates a combination of features with heaviest weight given the least desirable attributes. In this way the curve simulates assessment by a consistent unbiased observer. In the current investigation, however, the mathematical methods of the d-curve were set aside in favor of a simple sum and product combination of the ordinal categories, primarily because of the large quantity of data to be evaluated. Although the d-curve is more rigorous, significant deviations from its predictions were not anticipated with the simpler technique.

I. Elevated Temperature Stability

The coating's resistance to the effects of elevated temperature exposure was determined by a thermogravimetric (TGA) analysis. That is, the weight of a coated beta titanium coupon was monitored as a function of exposure time, with weight loss (or gain) considered a measure of stability. In the test, a loss of stability is revealed as a reaction between the coating and the furnace atmosphere, or between the furnace atmosphere and the titanium alloy sample. In general, a certain amount of weight loss is expected as organic binders and some water of hydration are liberated during heating. Significant weight changes are those in excess of the expected ones. The TGA data are summarized in Table A-1.

In addition to the weight changes, coating thickness measurements in the green and fused conditions discloses an important piece of data, namely, the relative degree of densification on fusion. Despite difficulties inherent in this measurement (a micrometer capable of gaging in 0.0001 inch increments with a constant force anvil and dial indicating head is required, variations in coating thickness with position must be recognized, and a thermal expansion correction between the elevated temperature thickness and the room temperature condition can be significant), the data are quite useful. In isothermal processing, the fused thickness has a major influence on interfacial friction, accumulation tendencies, and ease of release. In view of the variability from coating to coating in densification as shown by the TGA data, it is apparent that this factor must be optimized for consistency in final thickness, and therefore in forging behavior.

Several coating formulations exhibited a thickness increase on fusing. This increase, presumably a result of gas evolution forming a stable foam, gradually diminished with exposure time. The effect of this on forging behavior is not known, and therefore, for the purposes of this program, a small thickness increase not associated with a significant weight loss or gain was considered acceptable.

Thickness changes were not measured for those coatings that did not fuse to glassy consistency such as the CFC varieties. Durability of these is generally achieved through organic binder additions. Once this resinous material is removed in preheat, the coating becomes very thin. Caution must be exercised in handling, the coatings are quite soft and compliant, and thickness measurements are difficult to interpret. Therefore, densification was not considered in evaluation of the controlled friction coatings.

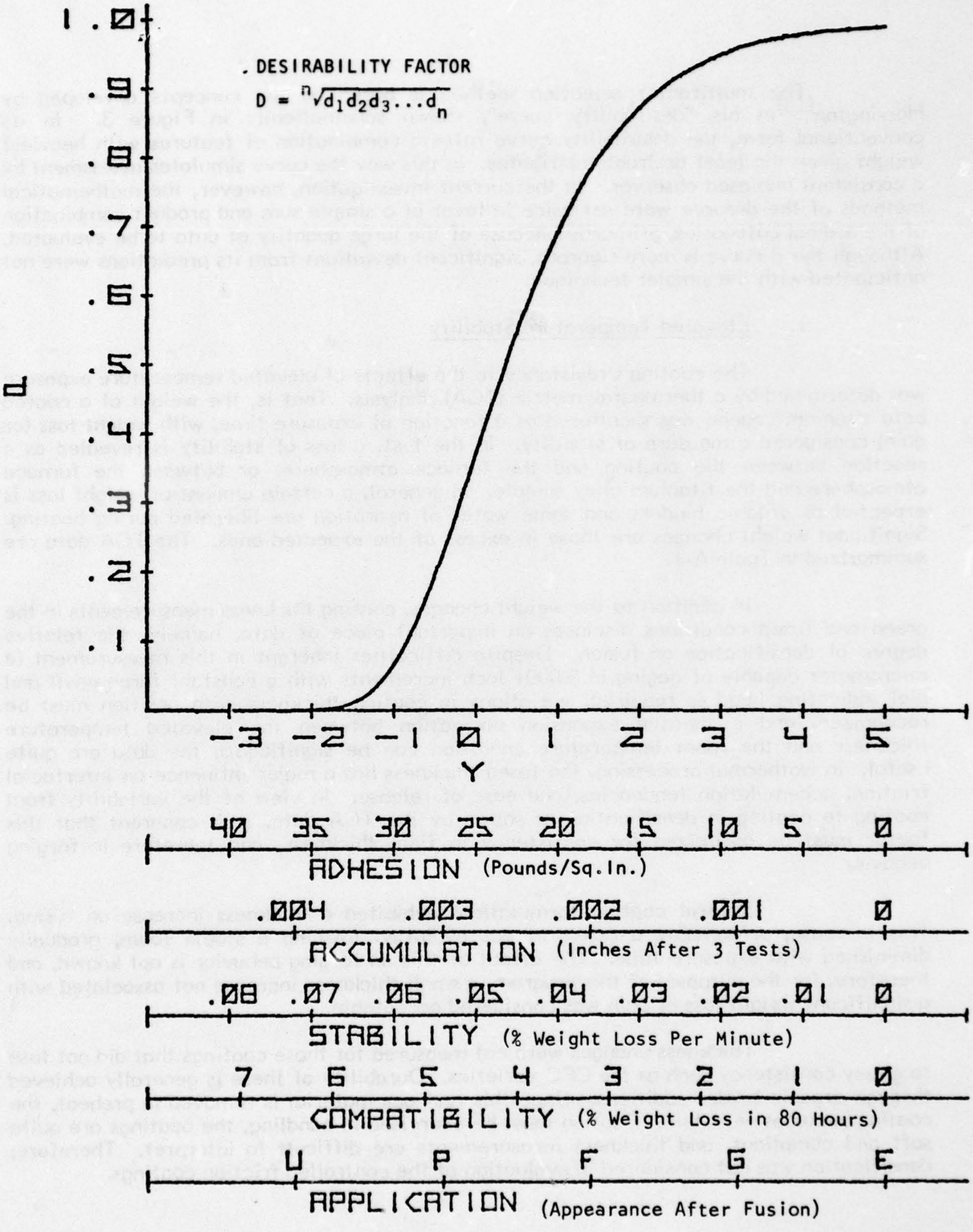


Figure 3. Graphical Method of Calculation For Desirability Factors

2. Coating-Die Material Compatibility

Data from the extended exposure compatibility evaluation are detailed in Table A-II. As anticipated, the chemical reaction rates were lowered significantly compared to the higher temperature work reported previously.[6] Nevertheless, compatibility breakdown continued to be an important effect with considerable variation among coating systems. A definite effect of particulate additions was observed, but the major contributor was the vitreous matrix itself. The oxides so effective in reducing viscosity to the levels required for low temperature isothermal processing, such as the alkalines and lead oxide, also demonstrate a reactive tendency.

The two die materials evaluated, namely A286 representing iron base alloys and IN100 representing nickel base alloys, behaved quite differently. A286 showed a high reaction rate when tested at 1500°F and above, but adequately withstood the oxide attack at 1350°F. Therefore coatings recommended for low temperature service are those that performed well with this alloy in view of its substantially lower cost. At 1500°F, however, only candidates compatible with IN100 are recommended.

Most prior experience with coating-die compatibility has shown a time dependent reaction as the important reaction mode. However, during this investigation, in the accumulation and adhesion evaluation program, some instances of localized pitting attack occurred during the relatively brief time period of the shear adhesion test. These events were few in number, very reproducible in multiple tests, and showed no apparent relationship to a compositional variable. The occurrence of this pitting attack was utilized as an evaluation factor in selection of recommended compositions. Evidence of pitting attack, either on the workpiece or the die material eliminated that composition from further consideration.

In the desirability index data of Table III, the extended exposure type compatibility was broken into five categories according to the relative reaction rate. Evidence of gross pitting was given a much more severe effect in that only three categories were selected, either no detectable attack, -1, very slight blemishing of the titanium alloy surface, -2, or noticeable pitting on the titanium alloy or die material.

3. Adhesion and Accumulation

Both factors were evaluated at one time with the shear adhesion apparatus described previously.[6] Adhesion is measured as the load (in psi) required to shear a film of a separation-lubrication substance one square inch in area and about 0.003-0.004 inches thick in the fused condition. In each test the film was first pressed between a beta C titanium alloy sample and a die material alloy sample with a load of approximately 3000 pounds. This load was determined to be just below the magnitude that caused measureable creep during the pressure application phase of the cycle. Compressive creep deformation of the A286 and IN100 was not measureable, and therefore the die material samples were reused throughout the program although the titanium pieces needed occasional redressing.

The accumulation was characterized by two parameters. Figure 4 illustrates these. The photograph shows die material samples from four shear adhesion tests after cooling from the test temperature. Two features are apparent on each sample. First, a uniform film of coating exists over about 1 square inch of the sample, and second, an extruded film of built-up coating remains at the edge where the two samples originally overlapped. The former condition, called "film accumulation" closely approximates the condition illustrated in Figure 2, namely the die conditioning and steady state film establishment. It can be measured quantitatively as the thickness of film remaining. The latter is a particularly bothersome form of accumulation, called "bulk accumulation." This is the material that is essentially extruded (in plane strain) between a deforming preform and the die surface. It is this type of accumulation that can lead to loss of detail at rib and projection extremities.

The four samples of Figure 4 are arranged in ascending order of "bulk" accumulation severity and thus illustrate the comparison standard utilized for the visual separation on a ranked scale for incorporation into the desirability assessment system. The category is essentially an estimate of the volume of material remaining.

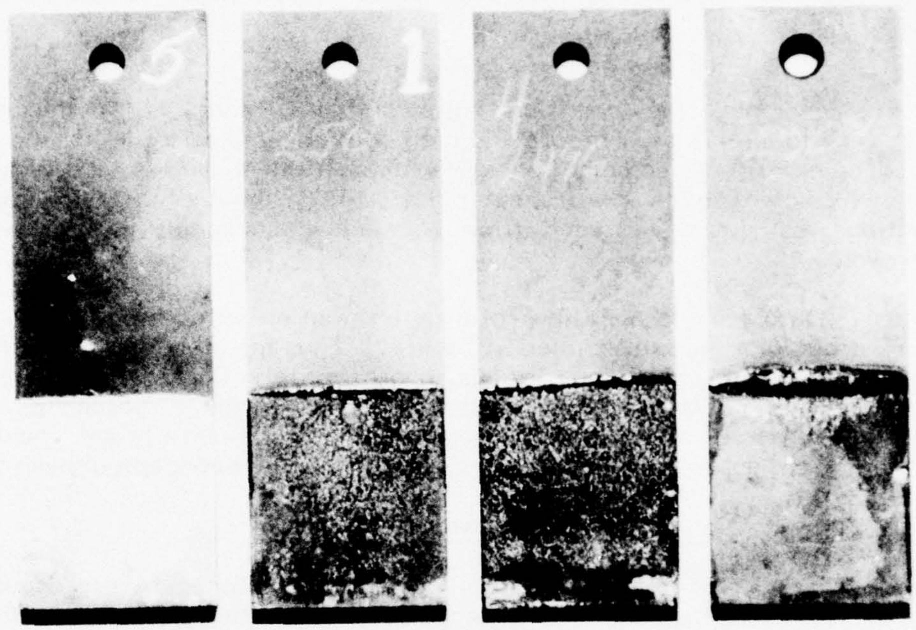
Results of the adhesion and accumulation tests are listed in Table A-III. In this table are recorded the adhesive shear stress, the "film" accumulation as mils of residual material in the uniform layer, the "bulk" accumulation volume class and the results of the visual examination for pitting attack.

The formulations examined with this test are those that looked promising based on application, stability and compatibility evaluation. As before, two die materials, A286 and IN100, were utilized, with the former evaluated at 1350°F and the latter at 1500°F. As discussed previously, a form of light pitting attack was noticed during these tests that did not necessarily correlate with the extended exposure compatibility data. This was more severe on A286 than on IN100 and virtually eliminated several compositions from further consideration.

4. The Partition Ratio

A quantitative determination of the partition ratio as illustrated conceptually in Figure 2 was performed. The procedure involved utilization of the shear adhesion evaluation performed in a somewhat different manner from that commonly employed. Normally, the adhesion test is carried out with successive coatings tested in shear between a titanium workpiece sample and a die material blank. Three shear stress measurements are performed sequentially using three different titanium alloy blanks coated to a thickness held constant from piece to piece. The die material blank is not cleaned after each exposure, but rather is allowed to accumulate the residual coating throughout the evaluation.

In a partition ratio measurement, a different procedure is required. Such procedure entails adhesion testing with removal of the die material sample for micrometer measurements after one exposure, cleaning and re-exposing for two trials, removal for measurement, and finally, cleaning and re-exposing for three consecutive trials. This series of six adhesion exposures thus provides an accurate measure of the rate of lubricant accumulation and aids interpretation of the normal adhesion procedure wherein the total accumulation after three exposures is noted.



1 2 3 4

Figure 4. Separation-Lubrication Accumulation Rating System.

The partition ratio data acquisition was complicated by the fact that the partition ratio model in Figure 2 applies to the "film" component of accumulation only, and does not easily account for bulk accumulation. This fact led to some unusual data combinations.

For example, the partition ratio and the steady film thickness were occasionally very low for substances having high bulk accumulation. This should not be misconstrued as a desirable combination. Even though the thickness remaining with the titanium alloy blank was small, it was greater than that on the die material sample. Most of the coating was simply extruded from between the samples during the pressure application cycle.

Data from a partition ratio determination on coating LFC 9, a variety with low bulk accumulation, are supplied in Table IV. Thus the steady film is estimated at 0.0009 inches and the partition ratio is approximately 0.2 (dimensionless). The experimental scatter to be associated with these data is high and a reasonable confidence band bracketing the true mean value of these parameters is quite broad. Nevertheless, the potential of the measurement technique is apparent and its conceptual basis is sound.

B. Separation-Lubrication Substance Selection

Results of the multi-factor optimization procedure are documented in Tables V and VI. These illustrate the individual evaluation ratings based on the data in Appendix A and the desirability categories of Table III. The tables also show the sum and product combinations of the individual ratings for both 1350°F service with A286 dies and 1500°F service with IN100. Final selection was made on the basis of a minimum sum, but when the sums are identical the higher product is preferred to insure a more uniformly satisfactory substance.

As has been discussed previously, the major coating types are identified by their code designations. That is, LFC designates a "low friction" coating; CFC, a "controlled friction" coating and MSC a "molten-salt" coating. In the LFC category, the particulates included nucleating agents, semi-abrasive particles and lamellar solids. The vitreous phases included three different compositions, namely,

1) 22A, a soft, lead oxide containing glass matrix with excellent viscosity characteristics at temperatures as low as 1350°F;

2) 38BB, a low viscosity borosilicate composition designed for low temperature service, made "meltable" by including potassium oxide as a fluxing agent; and,

3) a commercial formulation normally utilized as a low temperature extrusion lubricant supplied by Chi-Vit Corporation.

TABLE IV

Film Accumulation on A286 With Coating LFC9

	Trial 1	Trial 2
Single Exposure	0.0004	0.0006
Double Exposure	0.0008	0.0005
Triple Exposure	0.0009	0.0008

Initial Coating Thickness 3.2 mils per Side
in Fused Condition

Estimated Parameters

Steady Film Thickness - 0.0010

Partition Ratio - 0.2

TABLE V - COATING DESIRABILITY RATING

1350 F SERVICE - A286 DIES

	LFC7	LFC9	LFC11	LFC8	LFC10	LFC14	LFC16	LFC34
APPLICATION	2	2	1	3	1	1	1	1
STABILITY								
WEIGHT	5	4	3	1	2	3	2	1
THICKNESS	5	1	1	2	2	3	1	3
COMPATIBILITY	2	2	2	2	2	2	2	2
ADHESION	4	3	4	4	5	4	5	4
ACCUMULATION								
FILM	3	1	2	1	1	2	3	2
BULK	5	1	1	1	1	1	3	3
PITTING	1	1	1	1	1	1	5	1
SUM	27	15	15	15	15	17	22	17
PRODUCT	6000	48	48	48	40	144	900	144

TABLE VI - COATING DESIRABILITY RATING

1500 F SERVICE - IN 100 DIES

	LFC8	LFC7	LFC9	LFC11	LFC15	LFC17	LFC19	LFC34
APPLICATION	2	1	1	1	1	1	1	2
STABILITY								
WEIGHT	1	2	1	1	1	4	3	1
THICKNESS	2	1	2	1	1	1	3	3
COMPATIBILITY	3	2	2	2	2	3	4	3
ADHESION	3	4	1	3	3	5	3	5
ACCUMULATION								
FILM	3	3	2	2	3	3	2	3
BULK	3	1	1	1	3	1	3	5
PITTING	1	1	1	1	1	5	1	1
SUM	18	15	11	12	15	23	20	23
PRODUCT	324	48	8	36	54	900	648	1350

Compositions of the materials utilized including the matrix materials, particulates, carriers, binders, etc., are detailed in Table I. The formulations of the coatings are referenced in Table II. Note that this latter table does not include minor additives that might be considered such as suspension stabilizing agents or anti-foamants. These can be incorporated if needed in commercial usage, but in this program the suspensions were well enough behaved that neither settling nor foaming provided difficulty.

The coatings formulated in quantity for ring compression evaluation are described in the following paragraphs. Of the eight formulations, six represent the vitreous matrix-particulate type, and one each of the controlled friction and molten salt types were selected. Difficulty was encountered in formulating the latter types for stability and adherence during preheat. However, this difficulty does not outweigh the considerable potential afforded, especially in the CFC types, of achieving very fine surface finish on isothermal forging when the hot die systems are capable of supporting the relatively high loads involved.

1. Service at 1350°F - A286 Dies

a. LFC8

This coating is based on the 22A matrix phase with tantalum carbide as the refractory particulate phase. This coating was compatible with the iron base die material and showed good accumulation behavior, but did exhibit a slow weight loss when heated at 1350°F for a period of time.

b. LFC10

This coating also contains the 22A matrix which applies well to the beta titanium alloy. The semi abrasive particulate phase is chromium carbide at the 5.0 weight percent level in the fused coating. This formulation has proven excellent in application; superior in compatibility, stability and accumulation; but has demonstrated a somewhat high (although acceptable) adhesion load with this combination of die and workpiece alloy.

c. LFC34

This formulation utilizes DM254 as a matrix material with a fine dispersion of alumina as the particulate phase. This composition showed excellent application characteristics and was also strong on weight stability, compatibility and accumulation. It must be considered, at least for the present, as a lower temperature system (i.e., 1350°F) in view of its compatibility data on IN100 at 1500°F.

d. CFC10

This material consists of a mixture of graphite in sodium silicate. It represents the most successful "controlled friction" formulation from an application and adhesion standpoint. The material should again be regarded as a low temperature coating primarily for application to the dies, but one which can also be utilized on the workpiece.

e. MSC2

This system is based on a eutectic carbonate system commercially available from AF Holden Company designated as Holden 45. We have modified this material slightly by suspending it in alcohol and by the addition of 10% lithium carbonate. The purpose of the addition is to modify the composition so as to be somewhat off eutectic and thus partially solid at the forging temperature. Otherwise, the eutectic mixture has an extremely low viscosity and flows off the piece during transfer.

2. Service at 1500°F - IN100 Dies

a. LFC9

This formulation contains a borosilicate glass coded 38BB as the vitreous binder matrix phase, with tantalum carbide as the refractory particulate. This material performed well in all categories tested and should be an excellent wide-thermal-spectrum coating for beta alloy isothermal forging.

b. LFC15

Alumina was the particulate material in this formulation. The vitreous phase is 38BB. The mechanism of its action is not certain as it apparently dissolves fairly slowly into the vitreous matrix during preheat. Nevertheless, the coating rated well in most categories with a slightly higher but still acceptable shear adhesion strength.

c. LFC19

This formulation is the pure 38BB matrix ceramic. It is important that we establish a baseline with ring compression testing to determine the overall effect of the additives on friction behavior.

IV. CONCLUSIONS AND RECOMMENDATIONS

A program to develop and formulate separation-lubrication compositions for isothermal forging of beta titanium alloys in the temperature range from 1300°F to 1500°F was successfully completed. Several hundred compositions were chosen at the start of the program, mostly of the vitreous-phase-particulate type, containing boundary film additives or stable semi-abrasives. The field was narrowed to 28 low friction (LFC) types, 12 controlled friction (CFC) types and 8 molten salt (MSC) types. These were further evaluated through quantitative measurement of their application, fusion, stability, densification, accumulation and adhesion characteristics.

Optimum combinations of these factors were selected through a simplified multi-factor desirability analysis and eight final compositions were selected for further evaluation of their friction reducing properties through ring compression forging at the Westinghouse hot die isothermal forging facility at Wright-Patterson AFB. These compositions included three of the LFC type, one CFC type and one molten salt type for service at the low end of the temperature range (near 1350°F) on ferrous alloy dies, and three LFC type for service on nickel alloy dies at the high end of the thermal range (near 1500°F).

In general, interface control at these intermediate temperature levels involves several compromises. Generally, those vitreous ceramics that have sufficiently low viscosity to reduce friction significantly also contain elements that can cause pitting on titanium alloys or intergranular attack on nickel or iron base alloys. In the program, low viscosity in the LFC formulations was achieved according to a "forging window" concept with a lead oxide containing composition, a borosilicate-potassium glass and a high soda composition. It was anticipated that the relatively low service temperature would slow reaction rates to a degree that compatibility would not pose a problem. This did not prove to be the case however, as the sodium containing flux proved corrosive to both IN100 and A286.

Some recommendations based on the results of this program are as follows:

1) Separation-lubrication compositions developed on this program successfully meet stability compatibility and other conditions required for isothermal forging. Thus an investment in tooling and preforms has been reduced in risk.* Some further evaluation is advisable in terms of ring compression forging and in full scale component manufacture to establish friction factors and assess long term production effects. Feedback from these efforts will be valuable as verification of the desirability criteria utilized in this program.

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*Nevertheless, all substances supplied as a consequence of this program carry the following statement:

> "This coating has been tested for use in hot die isothermal forging. However, it is compounded solely for experimental purposes and in no event shall TRW be liable for special, indirect, or other damages including consequential damages arising in connection with the use of the material."

2) Composition of the vitreous phase component in low friction coatings is a critical variable in separation-lubrication coatings. Often the compositions are special purpose varieties that provide some difficulty in their production. Conversely, most inexpensive vitreous materials contain elements that reduce compatibility with nickel or iron alloy dies at high temperature. Clearly some compromise is required, and the techniques and standards established in this program should be adopted where possible as standards to gage future development.

3) Advanced concepts of separation-lubrication systems merit development, possibly on a fundamental scale. Some examples include pressure sensitive coatings whose viscosity becomes low under high pressure but remains high otherwise, and glassy coatings that react chemically with themselves to form a loosely adherent powder as a die residue after a certain residence time that exceeds that required for a forging operation.

4) Several die applied coatings developed on this program appear promising. These need further evaluation with additional development to reduce the associated costs. The controlled friction coating concept is an important one that will find application where as-forged surface finish is critical.

REFERENCES

1. Froes, F. H., Malone, R. F., Petersen, V. C., Rhodes, C. G., Chesnutt, J. C., and Williams, J. C., "Research on Deep Hardenable Titanium Alloy for Large Airframe Structural Members", Technical Report AFML-TR-75-41 on Contract F33615-71-C-1525, September 1975.

2. Gurney, F. J., Westinghouse Electric Company, 1975.

3. Vazquez, A. J., and Hayes, A. F., "Isothermal Forging of Reliable Structural Forgings", Ladish Company, Cudahy, Wisconsin, Final Report AFML-TR-74-123, AFML Contract F33615-71-C-1264, June 1974.

4. Vazquez, A. J. and Hayes, A. F., "Isothermal Forged Titanium Alloy Wheels", Interim Reports on AFML Contract F33615-75-C-5257, May 1975 through April 1976.

5. Spiegelberg, W. D., "Advanced Isothermal Forging Lubricants", TRW Inc. Final Engineering Report, Contract F33615-74-C-5011, Published with Phase I Wyman-Gordon Report;

   Chen, C. D., Couts, W. H., Gure, C. P., and Jain, S. C., "Advanced Isothermal Forging, Lubrication and Tooling Process", Wyman-Gordon Company, Contract F33615-74-C-5011, July 1974.

6. Spiegelberg, W. D., "Investigation of Separation-Lubrication Substance For Isothermal Forging", Phase III Task I Report to Westinghouse Electric Company, ER-7793, July 1976.

7. Doremus, R. H., <u>Glass Science</u>, John Wiley and Sons, New York, N. Y., 1973.

8. Morey, G. W., <u>The Properties of Glass</u>, Reinhold, New York, N. Y., 1954, 2nd Ed.

9. Shartris, T., and Spinner, S., "Viscosity and Density of Molten Optical Glasses", <u>Journal of Research of the National Bureau of Standards</u>, 46, 3, March 1951.

10. Kingery, W. D., <u>"Introduction to Ceramics"</u>, John Wiley & Sons, New York, 2nd Ed. 1975.

11. Kulkarni, K. M., Watmough, T., Staward, D., Parikh, N. M., "Isothermal Forging of Titanium Alloy Bulkheads", AFML-TR-74-138, August 1974.

12. Gurney, F. J., Jones, T. E., and Male, A. T., "Evaluation of Selected Commercial and Experimental Intermediate Temperature Forging Lubricants", AFML-TR-71-139, June 1971.

13. Harrington, E. C., "The Desirability Function", <u>Industrial Quality Control</u>, April 1965.

APPENDIX

EVALUATION DATA

ADVANCED LOW TEMPERATURE

SEPARATION-LUBRICATION SUBSTANCES

FOR

ISOTHERMAL FORGING

TABLE A-I

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1350°F Exposure	Before Exposure		After Exposure				
	Time (Min)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
D349-M	10	0.2632	0.0074	0.0949	0.0062	36.1	7.1	84.4
	20	0.2820	0.0080	0.1019	0.0069	36.1	7.7	86.2
LFC-1	10	0.4072	0.0073	0.0325	0.0251	8.0	2.5	345.5
	20	0.4318	0.0079	0.0349	0.0239	8.1	2.6	301.9
	30	0.4256		0.0374	NR	8.8	2.8	NR
	60	0.4236		0.0324	"	7.7	2.4	"
LFC-2	10	0.1950	0.0061	0.0401	0.0242	20.6	3.0	396.7
	20	0.2112	0.0054	0.0322	0.0238	15.3	2.4	439.8
	30	0.1945	0.0057	0.0291	0.0116	15.0	2.2	202.6
	60	0.1762	0.0058	0.0258	0.0068	14.6	1.9	117.4
LFC-3	10	0.4244	0.0070	0.0480	0.0038	11.3	3.6	54.0
	20	0.4473	0.0071	0.0501	0.0035	11.2	3.8	49.6
	30	0.4775	0.0078	0.0537	0.0038	11.3	4.0	49.0
	60	0.4763	0.0073	0.0532	0.0035	11.2	4.0	48.3
LFC-4	10	0.2349	0.0060	0.0351	0.0245	11.9	2.6	407.5
	20	0.2634	0.0061	0.0384	0.0540	11.6	2.9	885.2
	30	0.2511	0.0062	0.0365	0.0134	11.5	2.8	509.8
	60	0.2387	0.0068	0.0341	0.0343	11.3	2.6	504.4
LFC-5	10	0.2888	0.0077	0.0785	0.0103	27.2	5.9	133.1
	20	0.2726	0.0077	0.0796	0.0095	29.2	6.0	122.7
	30	0.3204	0.0076	0.0781	0.0077	24.4	5.9	100.7
	60	NR	0.0072	NR	0.0064			88.8

TABLE A-I (contd)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1350°F Exposure Time (Min)	Before Exposure Coating Weight (Grams)	Before Exposure Coating Thickness (Mils/Side)	After Exposure Coating Weight Loss (Grams)	After Exposure Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
LFC-6	10	0.1780	0.0043	0.0154	0.0058	8.7	1.2	133.7
	30	0.1974	0.0046	0.0171	0.0069	8.7	1.3	151.6
	60	0.1932	0.0044	0.0162	0.0057	8.4	1.2	129.9
LFC-7	10	0.2320	0.0075	0.0931	0.0130	40.1	7.0	173.3
	20	0.3185	0.0081	0.0942	0.0101	29.6	7.1	124.8
	30	0.2285	0.0080	0.0825	0.0110	36.1	6.2	136.9
	60	0.2402	0.0067	0.0322	0.0136	13.4	2.4	204.5
LFC-8	10	0.1827	0.0034	0.0155	0.0060	8.5	1.2	176.5
	30	0.1881	0.0034	0.0150	0.0029	8.0	1.1	83.8
	60	0.2047	0.0040	0.0166	0.0034	8.1	1.3	86.1
LFC-9	10	0.3111	0.0083	0.0411	0.0270	13.2	3.1	324.7
	20	0.3156	0.0085	0.0416	0.0199	13.2	3.1	234.9
	30	0.3149	0.0085	0.0415	0.0200	13.2	3.1	235.3
	60	0.2640	0.0071	0.0345	0.0090	13.1	2.6	127.0
LFC-10	10	0.3397	0.0060	0.0210	0.0029	6.2	2.9	48.3
	20	0.3094	0.0054	0.0195	0.0029	6.3	2.9	54.2
	30	0.3359	0.0056	0.0213	0.0030	6.3	3.0	52.7
	60	0.3676	0.0056	0.0240	0.0033	6.5	1.8	58.9
LFC-11	10	0.2932	0.0082	0.0390	0.0249	13.3	2.5	303.7
	20	0.2911	0.0087	0.0374	0.0208	12.9	2.1	240.5
	30	0.3071	0.0086	0.0402	0.0102	13.1	1.0	118.0
	60	0.2699	0.0069	0.0354	0.0078	13.1	2.7	113.1
LFC-12	10	0.4516	0.0077	0.0214	0.0092	4.7	1.6	120.3
	20	0.4152	0.0073	0.0195	0.0077	4.7	1.5	104.8
	30	NR	0.0065	NR	0.0067	NR	NR	102.3
	60	0.4117	0.0066	0.0210	0.0059	5.1	1.6	89.3

TABLE A-I (contd)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1350°F Exposure Time (Min)	Before Exposure Coating Weight (Grams)	Before Exposure Coating Thickness (Mils/Side)	After Exposure Coating Weight Loss (Grams)	After Exposure Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
LFC-13	10	0.2592	0.0078	0.0364	0.0283	14.0	2.7	365.2
	20	0.2630	0.0076	0.0366	0.0189	13.9	2.8	248.0
	30	0.2594	0.0077	0.0371	0.0187	14.3	2.8	242.9
	60	0.2489	0.0066	0.0334	0.0086	13.4	2.5	131.3
LFC-14	10	0.3188	0.0054	0.0191	0.0029	6.0	1.4	53.3
	20	0.3216	0.0051	0.0191	0.0030	6.0	1.4	58.8
	30	0.3398	0.0056	0.0206	0.0035	6.1	1.6	62.5
	60	0.4733	0.0084	0.0325	0.0040	6.9	2.5	47.6
LFC-15	10	NR	NR	NR	NR	NR	NR	NR
	20	"	"	"	"	"	"	"
	30	"	"	"	"	"	"	"
	60	0.3282	0.0093	0.0422	0.0146	12.9	3.2	156.5
LFC-16	10	0.3029	0.0048	0.0267	0.0030	8.8	2.0	61.5
	20	0.2997	0.0056	0.0257	0.0030	8.6	1.9	54.1
	30	0.3342	0.0058	0.0288	0.0043	8.6	2.2	73.9
	60	0.3375	0.0063	0.0313	0.0047	9.3	2.4	73.8
LFC-17	10	NR	NR	NR	NR	NR	NR	NR
	20	"	"	"	"	"	"	"
	30	"	"	"	"	"	"	"
	60	0.2985	0.0078	0.0380	0.0140	12.7	2.9	180.6

TABLE A-I (contd)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1350°F Exposure Time (Min)	Before Exposure Coating Weight (Grams)	Before Exposure Coating Thickness (Mils/Side)	After Exposure Coating Weight Loss (Grams)	After Exposure Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
LFC-3	10	0.6685	0.0086	0.0609	0.0036	9.1	4.6	41.5
	20	0.4613	0.0091	0.0611	0.0039	13.3	4.6	42.3
	30	0.4597	0.0086	0.0562	0.0037	12.2	4.2	42.7
	60	-	-	-	-	-	-	-
CFC-1	10	0.1248	0.0036	0.0620	0.0030	49.7	4.7	83.3
	20	0.1381	0.0038	0.0917	0.0030	66.4	6.9	78.7
	30	0.1858	-	0.1359	-	73.1	-	-
	60	0.1705	0.0041	0.1336	0.0035	78.4	10.1	86.4
CFC-2	10							
	20		NON-ADHERENT COATING					
	30							
	60							
CFC-3	10	0.4684	NR	0.0968	NR	20.7	7.3	NR
	20	0.5460	"	0.1147	"	21.0	8.6	"
	30	0.4818	"	0.1055	"	21.9	8.0	"
	60	0.3991	"	0.1143	"	28.6	8.6	"
CFC-4	10	0.1348	NR	0.1179	NR	87.5	8.9	NR
	20	0.1583	"	-	"	-	-	"
	30	0.1273	"	0.1047	"	82.3	7.9	"
	60	0.1763	"	0.1398	"	79.3	10.5	"

TABLE A-I (contd)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1350°F Exposure Time (Min)	Before Exposure Coating Weight (Grams)	Before Exposure Coating Thickness (Mils/Side)	After Exposure Coating Weight Loss (Grams)	After Exposure Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
CFC-5	10	0.4505	NR	0.3753	NR	83.3	28.3	NR
	20	0.3987	"	0.3287	"	82.4	24.8	"
	30	0.3477	"	0.2867	"	82.5	21.6	"
	60	0.3318	"	0.2729	"	82.3	20.6	"
CFC-6	10	0.3655	"	0.2731	"	74.7	20.6	"
	20	0.3106	"	0.2404	"	77.4	18.1	"
	30	0.3549	"	0.2710	"	76.4	20.4	"
	60	0.3586	"	0.2700	"	75.3	20.3	"
CFC-7	10	0.4519	NR	0.1121	NR	24.8	8.4	NR
	20	0.3073	"	0.0913	"	29.7	6.9	"
	30	0.3070	"	0.0886	"	28.9	6.7	"
	60	0.1850	"	0.0391	"	21.1	2.9	"
CFC-8	10	0.0837	NR	0.0180	NR	21.5	1.4	NR
	20	0.0724	"	0.0273	"	37.7	2.1	"
	30	0.0916	"	NR	"	NR	NR	"
	60	NR	"	"	"	"	"	"
CFC-9	10	COATING SPALLED SEVERELY						
	20							
	30							
	60							

TABLE A-1 (contd)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

	1500°F Exposure	Before Exposure		After Exposure				
Coating	Time (Min)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
LFC-7	10	0.4392	0.0042	0.0187	0.0191	4.3	1.4	454.8
	20	0.4140	0.0075	0.0168	0.0115	4.1	1.3	153.3
	30	0.3348	0.0064	0.0120	0.0074	3.6	.9	114.8
	60	0.2961	0.0100	0.0137	0.0069	4.6	1.0	69.3
LFC-9	10	0.3314	0.0060	0.0230	0.0056	6.9	1.7	93.3
	20	0.3674	0.0064	0.0253	0.0075	6.9	1.9	117.3
	30	0.3443	0.0068	0.0216	0.0087	6.3	1.6	127.9
	60	0.3577	0.0067	0.0232	0.0061	6.5	1.8	91.0
LFC-11	10	0.3344	0.0063	0.0199	0.0035	6.0	1.5	55.6
	20	0.3851	0.0067	0.0230	0.0046	6.0	1.7	67.9
	30	0.4019	0.0075	0.0213	0.0046	5.3	1.6	61.1
	60	0.3275	0.0073	0.0190	0.0045	5.8	1.4	61.0
LFC-10	10	0.2740	0.0071	0.0375	0.0096	13.7	2.8	135.5
	20	0.2953	0.0065	0.0371	0.0089	12.6	2.8	136.2
	30	0.2963	0.0081	0.0324	0.0090	10.9	2.4	111.8
	60	0.3275	0.0074	0.0190	0.0073	13.4	2.7	98.6
LFC-13	10	0.3941	0.0070	0.0176	0.0074	4.5	1.3	105.7
	20	0.2386	0.0033	0.0114	0.0029	4.8	.9	86.4
	30	0.4153	0.0063	0.0182	0.0052	4.4	1.4	82.4
	60	0.4282	0.0080	0.0203	0.0063	4.7	1.5	78.8
LFC-15	10	0.2924	0.0057	0.0169	0.0032	5.8	1.3	55.8
	20	0.3409	0.0061	0.0199	0.0033	5.8	1.5	54.1
	30	0.3801	0.0068	0.0204	0.0036	5.4	1.5	52.6
	60	0.2831	0.0052	0.0193	0.0032	6.8	1.5	61.2
LFC-17	10	0.3677	0.0068	0.0321	0.0034	8.7	2.4	50.4
	20	0.3783	0.0078	0.0340	0.0042	8.8	2.6	53.2
	30	0.3731	0.0033	0.0321	0.0042	8.6	2.4	127.3
	60	0.3447	0.0070	0.0328	0.0050	9.5	2.5	71.4

TABLE A-I (contd)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1500°F Exposure Time (Min)	Before Exposure Coating Weight (Grams)	Before Exposure Coating Thickness (Mils/Side)	After Exposure Coating Weight Loss (Grams)	After Exposure Coating Thickness (Mils/Side)	Weight Loss (%)	Weight Loss (mg/cm^2)	Thickness Ratio Fused/Green (%)
LFC-32	10	0.1940	0.0041	0.0149	0.0033	7.7	1.1	80.5
	30	0.1987	0.0043	0.0137	0.0048	7.0	1.1	111.6
	60	0.1989	0.0039	0.0132	0.0041	6.6	1.0	103.8
LFC-34	10	0.2305	0.0050	0.0183	0.0024	7.9	1.4	48.5
	30	0.2337	0.0054	0.0184	0.0024	7.9	1.4	43.9
	60	0.2307	0.0054	0.0177	0.0023	7.7	1.3	41.7
LFC-36	10	0.2443	0.0047	0.0189	0.0031	7.7	1.4	64.9
	30	0.2309	0.0056	0.0192	0.0030	8.3	1.5	53.6
	60	0.2325	0.0052	0.0182	0.0027	7.8	1.4	51.9
LFC-26	10	0.1895	0.0039	0.0087	0.0035	4.6	.7	89.7
	30	0.1776	0.0038	0.0056	0.0042	3.2	.4	• 112.0
	60	0.1776	0.0038	0.0042	0.0038	2.4	.3	98.7
LFC-28	10	0.1782	0.0036	0.0121	0.0025	6.8	.9	68.1
	30	0.1832	0.0036	0.0092	0.0044	5.0	.7	120.8
	60	0.1860	0.0037	0.0082	0.0044	4.4	.6	120.5
LFC-30	10	0.1874	0.0072	0.0145	0.0070	7.7	1.1	97.0
	30	0.1872	0.0039	0.0122	0.0146	6.5	.9	379.2
	60	0.2085	0.0039	0.0117	0.0168	5.6	.9	435.1

TABLE A-11

COATING VS. DIE MATERIAL - COMPATIBILITY

Temperature (°F)	Die Material	Exposure Time (Hours)	LFC 6	LFC 8	LFC 10	LFC 12	LFC 14	LFC 16
1350	A286	16	.18	.41	.35	.36	.43	.47
		32	.72	.75	.63	.75	.86	.89
		48	1.02	1.11	.90	1.13	1.22	1.27
		64	1.15	1.26	1.05	1.28	1.37	1.46
		80	1.27	1.39	1.15	1.50	1.54	1.59
			LFC 19	LFC 7	LFC 9	LFC 11	LFC 18	LFC 26
1350	A286	16	.39	.35	.36	.31	.35	.16
		32	.72	.69	.76	.77	.80	.36
		48	1.07	1.12	1.23	1.13	1.35	.59
		64	1.20	1.50	1.67	1.55	1.73	1.08
		80	1.31	1.80	1.92	1.77	2.03	1.63
1350	A286		LFC 28	LFC 30	LFC 32	LFC 34	LFC 36	LFC 38
		16	.24	.34	.24	.19	.47	.15
		32	.44	.59	.39	.42	.77	.34
		48	.67	.83	.50	.48	1.04	.40
		64	.92	1.30	.78	.78	1.44	.68
		80	1.53	1.82	1.26	1.36	2.07	1.27

TABLE A-11 (contd)

COATING VS. DIE MATERIAL - COMPATIBILITY

Temperature (°F)	Die Material	Exposure Time (Hours)	CFC 3	CFC 6	CFC 11	CFC 10	MSC 1	MSC 6
1350	A286	16	.15	.16	.16	.12	.15	.58
		32	.50	.51	.53	.56	.50	2.32
		48	.99	.98	1.15	.95	1.13	3.57
		64	1.38	1.36	1.53	1.28	1.52	4.50
		80	1.88	1.79	1.92	1.89	2.07	5.58
			LFC 18	LFC 20	LFC 22			
1350	IN 100	16	.32	.32	.31			
		32	.71	.69	.69			
		48	1.16	.90	1.08			
		64	1.77	1.49	1.68			
		80	2.16	1.83	2.12			

TABLE A-11 (contd)

COATING VS. DIE MATERIAL - COMPATIBILITY*

Temperature (°F)	Die Material	Exposure Time (Hours)	LFC 7	LFC 9	LFC 11	LFC 13	LFC 15	LFC 17
1500	IN 100	16	.13	.23	.20	.24	.16	.28
		32	.35	.52	.47	.66	.35	.62
		48	.62	.89	.76	1.17	.69	1.01
		64	1.10	1.40	1.27	1.77	1.20	1.60
		80	1.53	1.87	1.73	2.34	1.59	2.22
			LFC 18	**LFC 6**	**LFC 8**	**LFC 10**	**LFC 12**	**LFC 14**
1500	IN 100	16	.34	.42	.27	.19	.61	.57
		32	.68	.76	.44	.35	.90	.76
		48	1.06	1.52	1.24	.86	1.82	1.62
		64	1.49	2.53	2.48	2.08	3.04	3.01
		80	1.86	3.24	3.24	2.94	3.77	3.81
			LFC-19	**LFC 20**	**LFC 22**	**MSC-7**	**MSC-8**	
1500	IN 100	16	.40	.73	.73	.22	.18	
		32	.50	1.48	1.42	.40	.36	
		48	.86	2.10	2.05	**	**	
		64	2.19	2.74	2.63			
		80	3.01	3.27	3.17			
			CFC 3	**CFC 6**	**CFC 11**	**CFC 10**	**MSC-1**	**MSC-6**
1500	IN 100	16	.71	.54	7.85	.45	.63	.69
		32	1.77	1.50	8.77	1.42	2.87	2.85
		48	2.55	2.30	9.46	2.28	3.36	4.77
		64	3.66	3.22	10.23	3.19	5.25	5.13
		80	4.69	4.13	11.00	4.09	6.66	6.20

* Table entries are cumulative weight losses expressed as percentage of sample weight

**Material "foamed" badly, test results inaccurate, test discontinued.

TABLE A-III

ADHESION AND ACCUMULATION

1350°F Beta C on A286

Coating		Applied Load* (Lbs)	Green Coating Thickness (Mils/Side)	Shear Stress* (PSI)	Die Accumulation** Film (Mils)	Die Accumulation** Bulk	Die Accumulation** Pitting
LFC	7	3300	6.0	20.3	2.0	4	0
	9	3130	6.0	17.2	0.9	1	0
	11	2850	6.0	21.6	1.4	1	0
	8	3020	6.0	21.4	0.3	1	0
	10	3030	6.0	38.9	0.7	1	A286
	14	2990	6.0	26.6	1.8	1	0
	16	3050	6.0	31.2	2.1	2	A286
	34	3040	6.0	22.4	1.0	2	0
	38	3000	6.0	37.5	1.8	2	0
CFC	3	3040	6.0	21.4	16.7	1	A286, Ti
	6	3070	6.0	0	5.0	1	A286, Ti
	7	3040	6.0	5.4	0.4	1	A286
	10	3000	7.0	18.6	0.3	1	0
	11	3090	8.0	0	0.1	1	0
	12	3200	8.0	0	0.3	1	0
MSC	1	3110	7.0	0	1.8	2	A286
	2	3190	7.0	0	2.1	2	0
	3	3100	7.0	0	5.8	2	0
	4	3080	8.0	0	1.1	2	0
	5	3100	7.0	0	NR	NR	Ti

TABLE A-III (contd)

ADHESION AND ACCUMULATION

1500°F - Beta C on IN 100

Coating	Applied Load* (Lbs)	Green Coating Thickness (Mils/Side)	Shear Stress* (PSI)	Die Accumulation** Film (Mils)	Die Accumulation** Bulk	Die Accumulation** Pitting
GBTC 8	2940	6.0	21.5	1.9	1	0
LFC 7	2940	6.0	28.6	2.4	1	0
9	2990	6.0	9.7	1.9	1	0
11	3030	6.0	15.6	1.8	1	0
15	3030	6.0	18.2	2.1	2	0
17	3080	6.0	36.2	2.7	1	IN-100
19	3080	6.0	16.1	1.0	2	0
LFC 6	3030	6.0	23.2	1.0	2	0
8	3020	6.0	18.3	0.6	3	IN-100
10	3010	6.0	19.2	2.5	2	0
12	3020	6.0	27.9	0.7	2	0
14	3080	6.0	27.1	1.2	2	0
LFC 34	3020	6.0	36.0	2.4	3	0
38	3070	6.0	27.6	2.6	2	0

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